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(54) Title: CONSTRUCTION MATERIAL COMPRISING RECYCLED PVB (POLYVINYL BUTYRAL)

(57) Abstract: According to the invention, a construction material is disclosed comprising recycled PVB (polyvinyl butyral), one or more thermoplastic component(s) other than PVB, and cellulose-based material. Said cellulose material may in another embodiment of the invention also comprise a lignin component. In another embodiment of the invention, said construction material also comprises a glass particle component, and that said glass particle component is present in the form of particles or dust associated with the PVB. A further object of the invention is to provide a construction material comprising recycled PVB containing particles. With the construction material according to the invention, a field of use for PVB waste is provided, which at the same time can help solving the problem of the storage capacity of the increasing stocks of PVB waste.

CONSTRUCTION MATERIAL COMPRISING RECYCLED PVB (POLYVINYL BUTYRAL)

Field of the invention

This invention relates to a construction material comprising recycled PVB, one or more thermoplastic component(s) other than PVB, and cellulose-based material.

Background of the invention

PVB (polyvinylbutyral) is a polymer material having a chemical composition without any harmful substances, so that burning of the material may take place without giving rise to any particular problems. PVB film does not contain additives classified as dangerous or harmful.

PVB is primarily used as a laminated film in wind-screens in all modern vehicles such as cars and trucks. The PVB film prevents glass from splintering when damaged in a crash, etc. In some cases, PVB is included in glazing in buildings, especially in areas with hurricanes, or in safety windows.

The Michigan Molecular Institute has for several years tried to find applications for the PVB waste on assignment from the automotive industry in the USA and Du Pont®, one of 3 large PVB-producers in the world. A publication covering this topic has been issued.

New applications of recycled PVB has been developed, e.g., as insulation material for cables, in flooring application (Hoechst®), but none of these applications seems to have been a commercial success. Recycled PVB has also been tested for use as an additive in thermoplastic road marking materials. PVB also adheres well to glass, and a market for PVB may for this reason be present in this field in the future.

In Japan, the only Japanese PVB manufacturer, Sekisui®, has for some years attempted to find fields of use for PVB being recycled from the automotive industry. Apparently, Sekisui® has ended their project as no economically feasible field of use for recycled PVB has been discovered.

Unofficial figures state that in the USA, approximately

10 000 tons of PVB waste is generated per year from condemned vehicles. The total volume of recycled PVB is estimated to be between 40 000 and 75 000 tons, increasing by minimum 4-6% annually the next 10 years. Similarly, in Europe and Japan, volumes of 6 000 - 10 000 metric tons are generated per year.

Prior to the disclosure of the present invention, the only economically and practically feasible way of reducing the stocks of PVB waste has been through incineration of waste and recycling of energy. PVB carries a high amount of chemically bound energy. Less harmful fumes are released when burning PVB, than when burning heating oil. However, the price of energy recovered from such a waste product is sometimes lower than the price of energy recovered from oil, as bales of PVB waste are difficult to handle and large incinerators are needed in order to recover the energy.

Large amounts of PVB waste are stored world wide, which are very reasonable priced due to the large surplus and few applications for PVB. Most of the PVB waste contains some glass particles, which make the use more difficult. A lot of scrap plastic dealers are today selling recycled PVB-film in bales at very low prices.

Outdoor storage of PVB material is not recommended, as moisture and ultraviolet radiation will degrade the PVB waste. In addition, the PVB material will easily dirty when stored outdoors.

Because of the sticky and soft surface of the PVB film waste, glass particles, dust and other impurities easily stick to the surface and reduce the quality and the fields of use of the recycled product.

The storing capacity in many countries is limited. Accordingly, some of the problems with PVB waste are increasing storage costs and decreasing quality of the PVB waste due to degradation and impurities.

The automotive industry and manufacturers of PVB are trying to find economical and relevant fields of uses for the waste generated from the manufacturing processes of these industries. In some countries, such as Germany, all industry by law have to take care of the waste their

products generate in the whole life cycle. This means that the manufacturer is responsible for handling all waste created by the product from packaging to the recycled components of the product after being disposed of.

In fact, few economically or practically feasible fields of uses have been discovered for material of PVB, which may contain glass particles, pure or mixed with other materials. PVB film for use in windscreens is designed to prevent glass from splintering. Modern equipment for automatic separation of glass and PVB film has been developed, so that most of the glass can be recycled into new products. However, fragments of glass remaining in the PVB material may be inevitable, and complete separation of glass and PVB film seems not possible to achieve. In prior art, no solutions have been provided for using recycled PVB film containing particles.

Other recycled bulk- and special polymers like, e.g., polyethylene, polypropylene, polyvinyl chloride, polycarbonate and polystyrene, has successfully been included in construction elements such as window profiles, decks, fences and vehicle components. Frequently, the recycled thermoplastics are compounded with virgin material and with reinforcing additives or fillers like glass or wood fibres. Recycled materials are also used in insulation foams and other construction materials.

Composites of wood fibre and plastics (plastic lumber/-composite wood) have also been increasingly utilised by the automotive industry for vehicle components, e.g., as interior details such as hat shelves and door panels. Thermoplastic composites comprising cellulose-based materials like wood fibres, etc. based on PE, PP and PVC have been widely used in the USA for 2-3 years. Substitution of materials used in outdoor products such as decks, yacht harbours, fences, window frames and playgrounds will become more widespread. Today, common materials used for such products are different types of plastic, hard types of wood, and impregnated wooden material (such material often includes poisonous substances such as arsenic or arsenic compounds). The latter materials are environmentally

problematic because of pollution/poison problems and the forest industry having exploited the tropical forests for hard wood world wide too heavily. This is the main reason why plastic lumber decking products in the US in 2-3 years have increased from very small up to 3-400 000 tons per year (1999).

The automotive industry world wide has for some years developed interior details made of a mixture of poly-propylene and wood fibres / cellulose based fibres, and today a lot of applications have been found in modern vehicles world wide. Examples of such interior details are interior door panels, hat shelves and different hidden covers made of a polypropylen/wooden material composite.

Prior art

US 3.888.810 discloses a thermoplastic resin composition comprising one or more thermoplastic resins selected from the group consisting of polyethylene, polypropylene, polystyrene, polyvinyl chloride and acrylonitrile-styrenebutadiene resin, one or more wood materials selected from the group consisting of saw-dust, bark-dust and paper-mill wastes, and fibrous materials selected from the group consisting of polyester fibre, polyamide fiber, polyacrylic fibre, glass fibre, silica-alumina fibre and rayon.

In US 4.003.866, a construction material is described which comprises a mixture of a plastic component constituting at least one thermoplastic resin, and a filler component consitituting at least one mineral and/or organic filler in an amount at least equal in weight to the amount of said plastic component, the individual particles of the filler being coated or encapsulated with a polyethylene or polypropylene wax having a molecular weight of from 1,000 to 10,000, a thermofluid high molecular weight polymer, or a silicate coating material.

In US 5,194.461, a polymeric composition is described comprising an intimate mixture of chopped herbaceous fibers and granulated, previously-used high density polyethylene, heated to a moldable state and shaped into structural

components, said herbaceous fibers being from the group consisting of oat straw, soybean straw and corn stalk fibers.

A process for forming structural components from filled polymeric composite is also disclosed, including the steps of chopping herbaceous fibers into short lengths, the herbaceous fibers being from the group consisting of oat straw, soybean straw and corn stalk fibers, chopping previously-used polyethylene materials into small chips, intimately mixing the chopped herbaceous fibers and the chopped polyethylene chips, heating the mixed fibers and polyethylene chips into a moldable state, solidifying the moldable mixture into a composite building component.

US 5.759.680 discloses a composite extruded profile comprising from about 30 weight percent to about 70 weight percent of an unfoamed continuous phase of cleaned and classified recycled thermoplastic polymeric material consisting essentially of polyethylene and from about 70 to about 30 weight percent of a discontinuous phase of cellulosic fiber particles encapsulated in said thermoplastic polymeric material, wherein the encapsulation is the result of thorough dispersion of the cellulosic fiber particles within the polymeric material at a temperature above the softening point of the polymeric material and below a temperature where thermal degradation of the polymeric material or cellulosic fiber particles occurs.

A process of producing an agglomerate of wood waste and plastic is disclosed in WO 90/08020. According to WO 90/08020, the plastic is decomposed into smaller fractions, or is supplied in a decomposed state, and then the decomposed plastic is comminuted while being subjected to frictional heat. Further, moist wood waste is added during said comminution so that a mixture is formed. Thereafter, the mixture is agglomerated and recovered.

In EP 0.031.745, a composite material is described which is made up of wood dispersed in a thermoplastic material forming a matrix. The wood is present in the said material in a proportion of 15 to 50 % by weight relative to the final material in the form of fibrous particles of mean

diameter of between approximately 0.1 and approximately 1 mm and not exceeding 10 times the said diameter in length. The final material has a density which is from 10 to 20 % lower than the density of the thermoplastic material forming the matrix. In addition, a process for preparing the said material is described.

None of these publications show a construction material comprising recycled PVB. Furthermore, no solutions disclosed in the prior art has been provided for using recycled PVB film containing particles. The present invention makes use of a PVB waste material which now finds very few uses, if any at all.

Summary of the invention

According to the invention, a construction material is disclosed comprising recycled PVB (polyvinyl butyral), one or more thermoplastic component(s) other than PVB, and cellulose-based material. Said cellulose material may in another embodiment of the invenion also comprise a lignin component.

In another embodiment of the invention, said construction material also comprises a glass particle component, and that said glass particle component is present in the form of particles or dust associated with the PVB.

A further object of the invention is to provide a construction material comprising recycled PVB containing particles. With the construction material according to the invention, a field of use for PVB waste is provided, which at the same time can help solving the problem of the storage capacity of the increasing stocks of PVB waste.

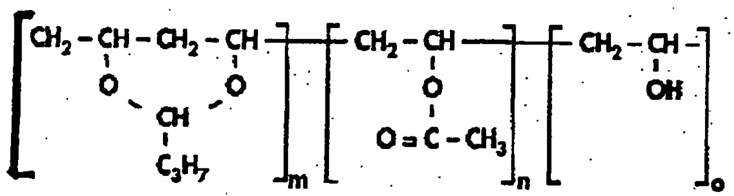
Detailed description of the invention

PVB (polyvinyl butyral) is a generic term of a group of plasticized polymers, which, in general, are manufactured through a reaction between a polyvinyl alcohol and a butyl aldehyde. The structural formula will vary dependent among other things on the conditions under which the reaction takes place.

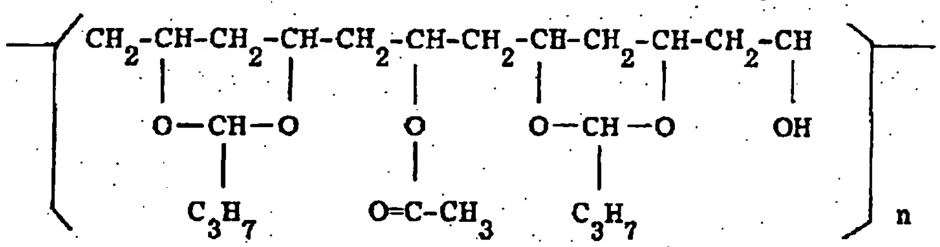
This group of polymers is supplied under several trade names, such as Butacite® from Du Pont®, Mowital® from Hoechst® and F-LEC B® from Sekisui®.

Below follows the structural formula of Mowital® (I) and F-LEC B® (II):

(I)



(II)



According to the invention, a construction material is disclosed comprising the following components: recycled PVB (polyvinyl butyral) one or more thermoplastic component(s) other than PVB, and cellulose-based material.

By adding recycled PVB to construction materials comprising thermoplastic and cellulose-based components, the following advantages can be obtained:

- lower price, as use of PVB is more cost effective than use of virgin and recycled polyolefins and PVC,
- better flow properties of the material and that the material becomes more easily workable because of the low melt viscosity of PVB,
- the possibility to load more cheap wood flour into the composite material to obtain a stiffer construction material, which
- lower weight of the product with more wood fibres,
- better flexibility and low temperature properties of the composite.

Said thermoplastic component(s) are preferably chosen from the group including recycled polyetylen (PE) of any kind such as LDPE, HDPE, LLDPE, MDPE and UHMWPE, polypropylen (PP) as a copolymer or homopolymer, and rigid or plasticized polyvinyl chloride (PVC).

Said cellulose based material may also comprise a lignin component. Preferably, said cellulose based material is chosen from the group including saw dust, wood flour, wood fibres, wooden chips or shavings, plant fibres such as straw, in any form, recycled paper and cardboard, and PE-coated cardboard boxes.

Depending on the amount of PVB in relation to other components comprised by the construction material, the stiffness of the construction material will vary. Increasing the amount of PVB in relation to other components comprised by the construction material will somewhat reduce the stiffness of the construction material.

Preferably, the amount of the thermoplastic component(s) included in the construction material is from 1 weight% to 70 weight% of the total weight of the construction material.

In another embodiment of the invention, the amount of said cellulose-based material included in said construction material is from 20 weight% to 90 weight% of the total weight of said construction material.

The amount of said PVB component included in said construction material is preferably from 0,5 weight% to 25 weight% of the total weight of the construction material. A construction material having a relatively high content of PVB is suitable as filler material and for construction elements where impact absorbing properties is important, in bumpers for cars.

In another preferred embodiment of the invention, the amount of said PVB component included in said construction material is from 2 weight% to 5 weight% of the total weight of said construction material. Adding PVB to constitue 2%-5% of the total weight of the construction material gives a construction material having a relatively low ability to absorb humidity, and a relatively high stiffness, which is suitable for construction elements, such as, e.g., planks, plates, joists, and similar.

The PVB component may include plasticizer(s) in an amount of up to 30 weight% of the total weight of the PVB component of the construction material. A construction

material comprising a plasticizer component has higher elastic properties.

Most PVB waste contains some glass particles, which make recovering and use of the PVB waste more difficult. Because surface of PVB film waste is sticky and soft, glass particles, dust and other impurities easily stick to the surface and reduce the quality and the fields of use of the recycled PVB material. Outdoor storage of PVB material is not recommended, as moisture and ultraviolet radiation will degrade the PVB waste. In addition, the PVB material will easily dirty when stored outdoors.

In a preferred embodiment of the invention, the construction material comprises a glass particle component, and said glass particle component is then present in the form of particles or dust associated with the PVB. Including this component into the construction material is clearly an advantage, as said gass particle component will reinforce the construction material. The amount of said glass particle component included in said construction material is preferably up to 8 weight% of the total weight of said construction material.

The construction material may also comprise grafted reactive polymer(s) and/or compatibilizer(s). A decrease in Young's modulus (E-modulus) of the construction material can be reduced or eliminated by including more fibres and/or by adding one or more types of compatibilizers. This leads to an improvement of the sound dampening effect and the low temperature properties of the construction material.

In a preferred embodiment of the invention, the amount of said grafted reactive polymer(s) and/or compatibilizer(s) included in said construction material is from 0,1 weight% to 5 weight% of the total weight of said construction material.

For increasing the toughness of the construction material, reinforcing fibres or particles such as glass fibres, carbon fibres, metal filaments or similar, and/or inorganic fillers, such as mineral sand or similar, may be a part of the composition of said construction material.

In another embodiment of the invention, the construc-

tion material comprise foam-forming agent(s). The result is a construction material having a relatively low density, which may, e.g., be used for manufacturing light-weight construction elements, such as interior details like hat shelves and door panels in cars.

Dependent on other desired properties, the construction material may also comprise one or more of the components selected from the group including lubricant(s), pigment(s), stabilizer(s), and flame retardant(s).

In the following, test procedures and test results will be presented and discussed.

EXAMPLE 1

Testing done by the inventors at Forschungsinstitutt für Kunststoffe und Recycling (FKuR) in Willich, Germany, which belongs to the Fraunhofer Institute in Düsseldorf, lead to the results which are described below.

7 test samples having compositons as described in the table 1 below were tested, named M1, M2,..., M6, M7. In the table 1, the amount of each component in each of the test samples is indicated in weight% of the total weight of each test sample. The melt index of the high density polyetylen used in the test sample was 2,9.

Component	M1	M2	M3	M4	M5	M6	M7
Wood flour	50	55	55	55	57	55	55
Stearic acid	-	-	-	3	-	1,5	-
Maleinated wax	1	1	1	-	-	1,5	-
PVB	15	10	14	12	15	12	5
Recycled HDPE MI 2,9	34	33	29	30	28	30	40
Foam-forming agent, Hostatron	-	1	1	-	-	-	-

Table 1

All mixtures were extruded on a Leitriz ZSE 40 GL single screw extruder with a 40 mm 36 D screw at 80 rpm at temperatures from 140 to 200 °C at the nozzle for all mixtures.

All components were mixed in a dry state before being fed into a hopper and the melted materials were extruded into flat profiles of 110x12 mm and cut into lengths of 250 mm which were annealed for 2 days and tested in a bending machine after DIN 53 452 with the following results:

Test/ (medium values)	**M1**	**M2**	**M3**	**M4**	**M5**	**M6**	**M7**
E-modulus (Young's modulus)/ stiffness N/mm^2	741	433	396	703	757	693	1154
3,5% Flexural stress N/mm^2	14,2	6,5	7,6	13,8	-	9,9	-
Bending strength N/mm^2	16,6	2,8	2,4	15,3	14,8	15,6	21,9
Flexural stress	8,33	4,00	3,78	7,66	7,74	7,97	11,56

Table 2

The results show that the mechanical properties are reduced when adding larger amounts PVB in relation to the other components. By using smaller wood particles the differences between mixes 1 and 6 would have been reduced. In oversized products wherein a large volume at a low cost is important and the mechanical properties are not critical, e.g. as a middle layer in a sewage pipe and substitutes for impregnated timber in sand boxes at playgrounds and energy absorbing components such as bumpers, the addition of at least 15% PVB could be included in the construction material without problems concerning processing properties.

The foam-forming agent seem to have a detrimental effect on the mechanical properties except the density of the total product, which was reduced by 15% as only 45% of the total weight of the test sample is foamable polymers.

For high performance products where high stiffness is of importance, such as, e.g., fences, structural car components and garden furniture, a level of PVB between 1% and 5% by weight will give the highest stiffness results. In particular, if PVB is introduced and melted onto the fibres before the rest of the binders are introduced into the mixture, this will lead to improved stiffness and reduced absorbtion of humidity.

Recycled PVB should preferably be mixed and melted together with cellulose-based material before a main binder and other additives, if any, are fed into a barrel in a mixing unit such as an extruder or compounding hot mixer. In this manner, the PVB will impregnate the fibres more thoroughly than when all components are mixed together in a dry state before being melted in the mixing unit.

Advantages of the construction material are the chemical structure and the extremely good flow of the material when heated in plastic machinery like extruders and calenders, together with the ability to wet cellulose based fibres.

The use of PVB as a wetting agent to impregnate cellulose-based material, with or without a content of lignin leads to the following effects:

- improvement of the adhesion between the cellulose material and one or more binders in the form of a poly-olefinic or vinyl-based polymer.
- reduction of the melt viscosity and improvment the flow of the polyolefinic- or vinyl-based polymer in order to improve the processabilty of the compound or make an increase of the cellulose material content possible. This will reduce the cost and specific gravity of the end product.
- compatibilizer effect, given appropriate conditions. The loose bound hydroxyl groups in the cellulose will be chemically bound to the polyvinyl butyral, which again will have a better adhesion to the polyolefinic-

or vinyl-based binder and therefore results in a more homogeneous product, as PVB has extremely good adhesion to most substrates.

Manufacturing of the construction material can take place using known plastic machinery such as extruders, injection moulding machines, mixers, 2 and 3-roll calenders and rotational moulding equipment.

When extruding or injection moulding the construction material, the PVB should preferably be premelted and mixed with cellulose material before one or more heated and melted polyolefinic or vinyl-based binders are mixed with the PVB-impregnated cellulose material.

Mixing of all components in a dry state before extrusion will lead to a reduction of the melt viscosity of the binder and to an improvement of the ductility of the mixture, so that the mixture becomes more easily workable, or make an increase in the level of said cellulose based materials possible.

For other non-screw plasticizing processing methods such as calendering, processing may be done using a completely dry compound/mixture. By impregnating said cellulose-based material with PVB in a separate operation before calendering, the adhesion effect will be improved.

The construction material can be further processed through, e.g., vacuum forming of sheets, milling, sawing, bending and drilling of semi-finished products such as sheets, rods and/or profiles.

The construction material according to the invention can advandageously be used as a substitute for existing construction materials which is environmentally harmful, e.g. impregnated wood, being utilised in constructions such as, e.g., decks, fences and window frames.

C l a i m s

1. Construction material c h a r a c t e r i z e d by comprising the following components:
- recycled PVB (polyvinyl butyral),
- one or more thermoplastic component(s) other than PVB, and
- cellulose-based material.

2. Construction material according to claim 1, c h a r a c t e r i z e d i n that said thermoplastic component(s) are chosen from the group including recycled polyetylen (PE) of any kind such as LDPE, HDPE, LLDPE, MDPE and UHMWPE, polypropylen (PP) as a copolymer or homo-polymer, and rigid or plasticized polyvinyl chloride (PVC).

3. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d i n that said cellulose material also comprises a lignin component.

4. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d i n that said cellulose-based material is chosen from the group including saw dust, wood flour, wood fibres, wooden chips or shavings, plant fibres such as straw in any form, recycled paper and cardboard, and PE-coated cardboard boxes.

5. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d in that the amount of the thermoplastic component(s) included in the construction material is from 1 weight% to 70 weight% of the total weight of the construction material.

6. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d in that the amount of said cellulose-based material included in said construction material is from 20 weight% to 90 weight% of

the total weight of said construction material.

7. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d in that the amount of said PVB component included in said construction material is from 0,5 weight% to 25 weight% of the total weight of the construction material.

8. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d in that the amount of said PVB component included in said construction material is from 2 weight% to 5 weight% of the total weight of said construction material.

9. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d in that said PVB component include plasticizer(s) in an amount of up to 30 weight% of the total weight of the PVB component of the construction material.

10. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d b y also comprising a glass particle component, and that said glass particle component is present in the form of particles or dust associated with the PVB.

11. Construction material according to claim 10, c h a r a c t e r i z e d in that the amount of said glass particle component included in said construction material is up to 8 weight% of the total weight of said construction material.

12. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d by also comprising grafted reactive polymer(s) and/or compatibilizer(s).

13. Construction material according to claim 12, c h a r a c t e r i z e d that the amount of said grafted reactive polymer(s) and/or compatibilizer(s) included in said construction material is from 0,1 weight% to 5 weight% of the total weight of said construction material.

14. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d by also comprising one or both of the following components:

- reinforcing fibres or particles, such as glass fibres, carbon fibres, metal filaments or similar, and
- inorganic fillers, such as mineral sand or similar.

15. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d by also comprising foam-forming agent(s).

16. Construction material according to anyone of the preceding claims, c h a r a c t e r i z e d by also comprising one or more of the following components:

- lubricant(s), pigment(s), stabilizer(s), and flame retardant(s).

17. Use of PVB as a wetting agent to impregnate cellulose-based material, with or without a content of lignin.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/NO 00/00314

A. CLASSIFICATION OF SUBJECT MATTER

IPC7: B27N 1/02, E04C 2/26 // C 08 L 29/04
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: B27N, C08J, C08L, E04C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5380794 A (R.E. SCHAEFER ET AL.), 10 January 1995 (10.01.95), abstract, claims --	1-16
Y	US 5759680 A (BROOKS JOE G. ET AL.), 2 June 1998 (02.06.98), abstract, claims --	1-16
X	DE 4229711 A1 (HüLS TROISDORF AG), 10 March 1994 (10.03.94), abstract, claims --	17
A	FR 2054564 A1 (GLAVERBEL), 23 April 1971 (23.04.71), claims --	1-16

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier application or patent but published on or after the international filing date
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- "&" document member of the same patent family

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